



Europäisches Patentamt
European Patent Office
Office européen des brevets



Publication number:

0 451 786 A2

(12)

EUROPEAN PATENT APPLICATION

(21) Application number: **91105606.7**

(51) Int. Cl.⁵: **D01F 6/62, D01F 6/70**

(22) Date of filing: **09.04.91**

(30) Priority: **13.04.90 JP 98087/90**

(43) Date of publication of application:
16.10.91 Bulletin 91/42

(84) Designated Contracting States:
DE FR GB IT NL

(71) Applicant: **DAI-ICHI KOGYO SEIYAKU CO., LTD.**

**55 Nishishichijo Higashikubo-cho
Shimogyo-ku Kyoto(JP)**

(72) Inventor: **Fujita, Takeshi**

**Ono 114-9, Hiranocho
Uji-city, Kyoto(JP)**

Inventor: **Isoda, Chuzo**
Shinmeimiyakita 64-9

Uji-city, Kyoto(JP)

Inventor: **Pu, Sejin**

**Kurumamichicho 6, Saga Tenryuji
Ukyo-ku, Kyoto(JP)**

(74) Representative: **Strehl, Schübel-Hopf,
Groening**

**Maximilianstrasse 54 Postfach 22 14 55
W-8000 München 22(DE)**

(54) **Water-soluble fiber and a method for manufacture thereof.**

(57) This invention relates to a water-soluble fiber and a method of manufacturing the fiber. The water-soluble fiber of the invention is composed of a high molecular compound with a weight average molecular weight of not less than 10,000, which high molecular compound is obtainable by reacting a polyalkylene oxide compound, which is obtainable by addition polymerization of an ethylene oxide-containing alkylene oxide and an organic compound containing two active hydrogen atoms, with a polycarboxylic acid, an anhydride thereof, a lower alkyl ester thereof, or a diisocyanate and is manufactured by spinning the same high molecular compound. The water-soluble fiber of the invention is not only high in elongation and tensile strength but is readily soluble in water.

EP 0 451 786 A2

BACKGROUND OF THE INVENTION

The present invention relates to water-soluble fiber and a method for manufacture thereof.

Among the water-soluble fibers heretofore known is polyvinyl alcohol fiber which is generally fabricated into a twisted yarn, woven fabric or non-woven fabric and put to use in a diversity of applications. For example, it has been used for provisional reinforcing or adhesion in a variety of industrial processes.

However, polyvinyl alcohol fiber is not readily soluble in water and requires heating to at least about 80°C for dissolution, thus being poor in workability. Moreover, when exposed to a temperature over 100°C, it undergoes crosslinking so that dissolution is often made difficult.

SUMMARY OF THE INVENTION

The object of the present invention is to provide a water-soluble fiber which is tough and readily soluble in water at ambient temperature and does not suffer losses in water solubility on exposure to heat and a method for manufacturing the fiber.

The present invention is directed to a water-soluble fiber composed of a high molecular compound with a weight average molecular weight of not less than 10,000, which high molecular compound is obtainable by reacting a polyalkylene oxide compound, which is obtainable by addition polymerization of an ethylene oxide-containing alkylene oxide and an organic compound containing two active hydrogen atoms, with a polycarboxylic acid, an anhydride thereof, or a lower alkyl ester thereof, or a diisocyanate compound.

The manufacture of said water-soluble fiber comprises spinning said high molecular compound.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

The polyalkylene oxide compound as a major starting material for the high molecular compound of the present invention is preferably a compound having a weight average molecular weight of not less than 100 and can be prepared by addition polymerization of an ethylene oxide-containing alkylene oxide and an organic compound containing two active hydrogen atoms.

The organic compound containing two active hydrogen atoms includes, *inter alia*, ethylene glycol, propylene glycol, polyethylene glycol, polypropylene glycol, polytetramethylene glycol, 1,6-hexanediol, bisphenol A, aniline and so on.

The ethylene oxide-containing alkylene oxide includes, *inter alia*, ethylene oxide and mixtures of

ethylene oxide with propylene oxide, butylene oxide, styrene oxide, α -olefin oxides, glycidyl ethers and so on.

The addition reaction of such an alkylene oxide can be carried out in the known manner, and the mode of addition polymerization of ethylene oxide and other alkylene oxides may be optionally random or block.

The polycarboxylic acid, anhydride thereof, or lower alkyl ester thereof, which is reacted with said polyalkylene oxide compound, include, *inter alia*, phthalic acid, isophthalic acid, terephthalic acid, sebacic acid, dimer acid, pyromellitic acid, etc., anhydrides thereof, and methyl esters, dimethyl esters, diethyl esters, etc. thereof. The preferred are dimethyl terephthalate, dimethyl phthalate, dimethyl isophthalate, dimethyl sebacate, pyromellitic anhydride and so on.

The polyester-forming reaction between said polyalkylene oxide compound and said polycarboxylic acid, anhydride thereof or lower alkyl ester thereof is preferably conducted at 120-250°C and 10^{-2} - 10^3 Pa.

The diisocyanate to be reacted with said polyalkylene oxide compound includes, *inter alia*, tolylene diisocyanate, diphenylmethane diisocyanate, hexamethylene diisocyanate, isophorone diisocyanate, xylylene diisocyanate, 4,4'-methylene-bis-(cyclohexyl isocyanate) and so on.

The urethane-forming reaction between said polyalkylene oxide compound and said diisocyanate is conducted by mixing the starting compounds in an NCO/OH ratio of, for example, 1.5 through 0.5 and heating the mixture at 80 to 150°C for 1 to 5 hours.

The weight average molecular weight of the high molecular compound according to the present invention is not less than 10,000. If the molecular weight is less than 10,000, the fiber will not be sufficiently high in mechanical strength, giving rise to yarn breakage in the spinning process.

This high molecular compound can be processed into fiber by any known relevant technique such as melt-spinning, dry spinning, wet-spinning, etc., although the melt-spinning process is preferred if only from economic points of view.

In the melt-spinning process, the high molecular compound is melted at 50-200°C in a nitrogen gas atmosphere and extruded from the conventional spinning nozzle.

In this process, such additives as a plasticizer, lubricant, stabilizer, colorant, filler, etc. can be added. According to the intended application, a perfume, fungicide, agrochemical, fertilizer, etc. can also be incorporated.

The water-soluble fiber as spun has large elongation. For example, the elongation of a fiber with a

diameter of 10 μm to 5 mm is approximately 500 to 3,000 percent.

After spinning, the fiber may be treated with a sizing or bundling agent in the hydrocarbon series or stretched. When it is stretched in a draw ratio of 5 to 30, its tensile strength is remarkably increased. The stretching may be performed concurrently with spinning.

The fiber thus obtained is subjected to various processings such as twisting and cutting. The fiber can also be constructed into mixed fabrics with other fibers.

The water-soluble fiber according to the present invention is not only high in elongation and tensile strength but is readily soluble in water. Moreover, this water solubility is not appreciably affected by heating. Therefore, as processed into a thread or yarn or a web, the fiber remains tough and self-supporting in application and, yet, can be completely dissolved out swiftly as needed. In this and other ways, this material can contribute to rationalization of various industrial processes.

The following examples and comparative examples are further illustrative of the invention, it being to be understood, however, that the invention is by no means limited thereof but limited only by the claims appended hereto.

Example 1

One hundred parts (parts by weight; the same applies hereinafter) of polyethylene glycol (weight average molecular weight 10,000) and 2.2 parts of dimethyl terephthalate were used to give a polyester compound with a weight average molecular weight of 130,000 (hereinafter referred to as high molecular compound A).

This high molecular compound A was fed to a melt-spinning apparatus, where it was melted at 120°C in a nitrogen gas atmosphere and extruded through a spinning nozzle at a rate of 50 m/min, rapidly cooled under tension and taken up. A plain-weave fabric was then constructed using a thread-like bundle of ten filaments.

When put in water, this plain-weave fabric disappeared in 15 seconds. On the other hand, the same plain-weave fabric was locally heat-sealed (120°C, 1.5 seconds) to provide a firm local bond. When this fabric was put in water, the whole fabric inclusive of the bond disappeared in 18 seconds.

Example 2

One-hundred parts of a polyalkylene oxide compound (weight average molecular weight 20,000) prepared by block polymerization of ethylene oxide (85%), propylene oxide (15%) and bisphenol A was mixed with 0.84 part of hexamethylene diisocyanate and a small amount of dibutyltin dilaurate and the mixture was heated at 100°C to give a high molecular compound with a weight average molecular weight of 250,000.

This compound was extruded in the same manner as Example 1, cut to 2-3 mm and processed into a nonwoven fabric. Using this nonwoven fabric as a filter, a used vacuum pump oil (containing 0.5% of water) was purified. As a result, the water content was reduced to 0.02% and the oil came clear. This purified oil could be reused with full competence.

When a plain-weave fabric of polyvinyl alcohol fiber was put in hot water at 80°C, it disappeared in about 10 seconds. In water at 25°C, however, the fabric swelled as its surface was wetted but remained to be dissolved completely as yet even after one hour. On the other hand, the same plain-weave fabric with a heat-sealed local bond remained partially undissolved even in hot water at 80°C.

Comparative Example 1

The high molecular compound A synthesized in Example 1 was fed to a melt-spinning apparatus, where it was melted at 120°C and extruded from a 1 mm (dia.) spinning nozzle at a take-up rate of 30 m/min. The resulting monofilament had a diameter of 40 μm and a tensile strength of 200 bar.

This fiber could be stretched in a draw ratio of 13 at 20°C and the stretched fiber had a diameter of 12 μm and a tensile strength of 390 bar.

At 50°C, the fiber could be stretched in a draw ratio of 20 to give a filament with a diameter of 9 μm . The tensile strength of this filament was 550 bar.

Comparative Example 2

A wet-spun polyvinyl alcohol filament with a diameter of 40 μm could be stretched only to twice its initial length at 20°C. The stretched filament had a diameter of 28 μm and a tensile strength of 500 bar.

It is, thus, apparent that in the case of the water-soluble fiber according to the present invention, its large elongation permits stretching into finer fiber even if the filament as spun is not so fine. Moreover, spinning troubles such as yarn breakage can be prevented in accordance with the invention.

Claims

1. A water-soluble fiber composed of a high molecular compound with a weight average molecular weight of not less than 10,000, which high molecular compound is obtainable by reacting a polyalkylene oxide compound, which is obtainable by addition polymerization of an ethylene oxide-containing alkylene oxide and an organic compound containing two active hydrogen atoms, with a polycarboxylic acid, an anhydride thereof, a lower alkyl ester thereof, or a diisocyanate.
2. A water-soluble fiber according to claim 1 which has an elongation of 500 to 3,000 percent.
3. A method of manufacturing a water-soluble fiber which comprises spinning a high molecular compound with a weight average molecular weight of not less than 10,000, which high molecular compound is obtainable by reacting a polyalkylene oxide compound, which is obtainable by addition polymerization of an ethylene oxide-containing alkylene oxide and an organic compound containing two active hydrogen atoms, with a polycarboxylic acid, an anhydride thereof, a lower alkyl ester thereof, or a diisocyanate.
4. A method of manufacturing a water-soluble fiber according to claim 3 wherein the fiber as spun or in the course of spinning is stretched in a draw ratio of 5 to 30.

35

40

45

50

55

4